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TECHNICAL REPORT T-79-76

IMPROVED SPECIFICATIONS FOR COMPOSITE
PROPELLANT BINDERS FOR ARMY WEAPON
SYSTEMS

James G. Carver
Technology Laboratory

27 July 1979



U.S. ARMY MISSILE COMMAND
Redstone Arsenal, Alabama 35809

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INTRODUCTION

Present analytical procedures for the analysis of polymeric binder materials for composite propellants are inadequate. They are time consuming, ineffective, difficult to accomplish and, for these reasons, often neglected by the propellant manufacturer. Very few specifications make any reference to molecular weight distribution. Recent experience with HAWK propellant is an example. Polymeric binders used in the production of several thousand HAWK motors at a cost of \$5000 each contained an insoluble fraction which varied significantly between lots of polymer. As a result of using this poor quality binder in the preparation of HAWK motors, it became necessary to reevaluate these motors and modify quality control procedures.

Since these motors represent a multimillion dollar cost, questions could have been avoided had the specifications called for molecular weight distribution analysis obtained either by gel permeation chromatography or by high pressure liquid chromatography. This instrumental method of analysis would add significantly to the specifications of all polymeric materials used in propellants. This includes the neo pentyl glycol azalate (NPGA), used in HAWK; hydroxy terminated polybutadiene (HTPB), used in VIPER, PATRIOT, Met Rocket, GSRS, PERSHING, HELLFIRE; polybutadiene acrylic acid (PBAA), used in PERSHING, SPRATAN; and polyglycol adipate (PGA), polycaprolactone (PCP), and polyethylene glycol (PEG), used in composite smokeless propellants presently under development.

The GPC and LC have both been used for a number of years for the characterization of polymers as those mentioned above. These polymers have all been studied using these instruments. Therefore, the basic data required and basic techniques necessary to involve these instruments in polymer specifications (molecular weight distribution) is available.

It only remained to develop a procedure and calibration technique that would be precise, accurate, and rapid enough to warrant its inclusion in the specifications.

RESULTS

Evaluation of Parameters

Before any work can be performed on calibrating a separation, all factors that effect that separation must be carefully evaluated and optimized. The first step is to determine which column or columns are necessary to obtain a good picture of the molecular weight distribution. That is, to broaden the samples peak in order to determine a representation of the amount of material in a series of narrow ranges of molecular weights. It is important for accuracy that those ranges that can be precisely measured be as small as possible and the peak as broad as possible.

If by broadening the peak, resolution is lost, then accuracy suffers. It can be seen from the following equation that the only way the peak width, w , can be increased without losing accuracy is by increasing V_i , the elution volume.

$$N = 16 \frac{(V_i)^2}{w}$$

This is accomplished in gel permeation chromatography by adding columns by which the molecules of polymer are partially retained. Separation in gel permeation chromatography is dependent, at least ideally, solely on molecular size and all material will elute from a column between V_o and V_t which are the void volume and the total permeation volume. A plot of V_i versus molecular size for any GPC column train will be similar to Figure 1. As V_i of any particular molecular size approaches either V_o or V_t , the linearity of the calibration is lost. An optimum selection of columns would allow the sample to elute over the entire linear range and not enter the nonlinear range. On this principle, the columns which offered potential separations were evaluated. Table 1 shows that the 100 Å column did little in separating the polymer. The 10^5 column was of limited assistance since the polymer eluted from it very close to V_t where solvent impurities elute. Of the two column combinations $500 + 10^4$ was the optimum. Even though the sample peaked well away from V_t , it was decided that further separating power in the low molecular size range was needed since derivation of the polymer (which would occur later) would add materials which eluted in that area. Therefore, the four column combination of

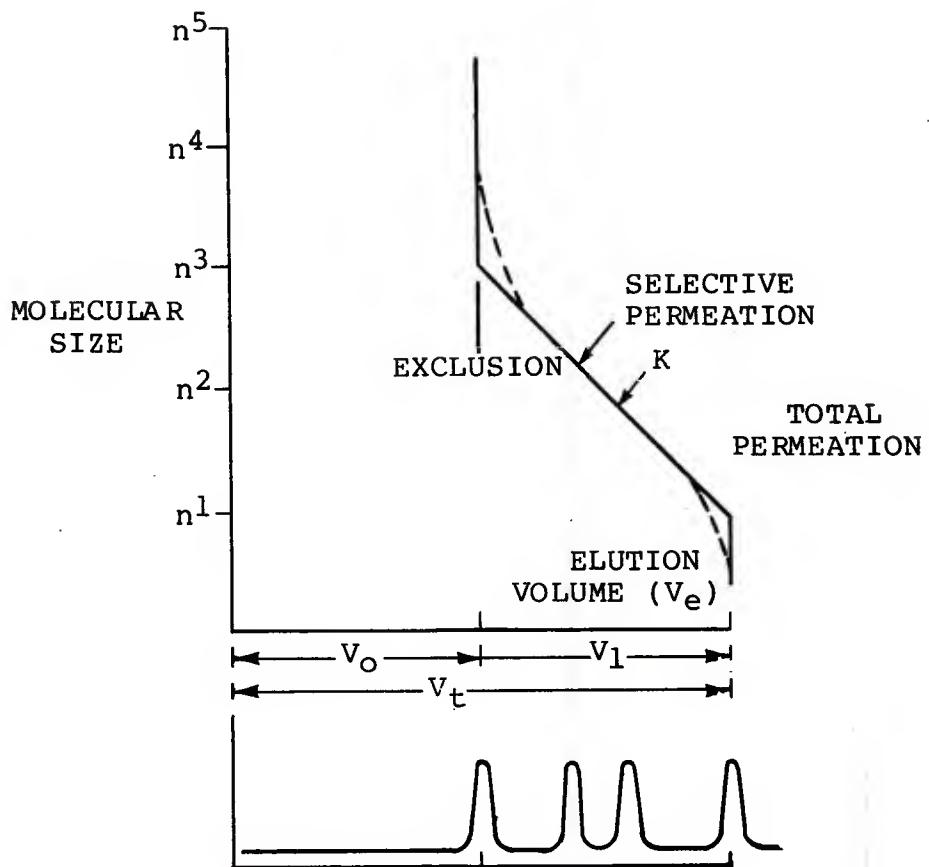


Figure 1. Elution volume versus molecular size for any GPC Train.

TABLE 1. K VALUES FOR R45M

Column (s)	K of HTPB-R45M Lot 303305
$10^5 \text{ } \text{\AA}$	0.77 (2 peaks)
$10^4 \text{ } \text{\AA}$	0.54
$500 \text{ } \text{\AA}$	0.17 (2 peaks)
$100 \text{ } \text{\AA}$	0.00
$10^4 + 10^5 \text{ } \text{\AA}$	0.69
$500 + 10^4 \text{ } \text{\AA}$	0.34
$100 + 500 \text{ } \text{\AA}$	0.20 (2 peaks)
$100 + 500 + 10^4 \text{ } \text{\AA}$	0.31
$500 + 10^4 + 10^5 \text{ } \text{\AA}$	0.43

$$K = \frac{V_i - V_o}{V_o}$$

$100 \text{ } \text{\AA} + 500 \text{ } \text{\AA} + 10^4 \text{ } \text{\AA} + 10^5 \text{ } \text{\AA}$ was selected for further work.

During the course of this project, the efficiency of the columns degraded due to nonexclusive use. A new column train was recalibrated using a $10^3 \text{ } \text{\AA}$ column which had been previously unavailable. The new column train consisted of $10^4 + 10^3 + 500 + 100 \text{ } \text{\AA}$.

Since in gel permeation chromatography, separation is accomplished solely by the column, it is only necessary that the solvent completely dissolve the polymer. Since it was planned that both refractive index and ultraviolet type detectors would eventually be employed, a further constraint was added that the solvent have a UV cut off close 200 nm. Many solvents would satisfy these requirements but, since almost all previous work with HTPB-R45M had been performed using tetrahydrofuran (THF), this solvent was arbitrarily selected.

It is well known that flow rate of the solvent has an effect on the resolution in gel permeation chromatography. This is maximized at a flow rate of about 0.9 ml per minute for the microstyrigel columns. Although separating power is diminished at higher flow rates, the effect is only

slight up to 2 ml per minute. Since the effect is slight and recalibration would only require a short time, a flow of 2.0 ml/minute was tentatively selected until later work would permit an evaluation of the trade offs of accuracy and speed.

The final two variables evaluated were sample concentration and sample volume. To maximize the detectability of a sample and thus the accuracy of the analysis, the maximum amount of material possible should be sampled. However, if the column is overloaded its separation capability will be impaired. If a large volume of sample is injected, a particular molecular size will elute over a broad volume rather than the narrow range required for accuracy. Since volume and concentration are related, both these parameters were evaluated simultaneously as the matrix in Figure 2 indicates. Thus, concentrations from 0.001 to 0.48 mg/ μ l of polymer and sample volumes from 1 μ l to 1 ml were evaluated. The limit of detectability on the RI was 0.05 mg per sample. For the 0.1 mg sample size, very little effect was seen on the peak width up to 100 μ l sample volume. For the 1.0 mg sample size, however, a significant broadening occurred between the 20 μ l and 100 μ l sample volume. When the volume was held constant at 10 μ l the value of K increases slightly but steadily for samples greater than 0.5 mg.

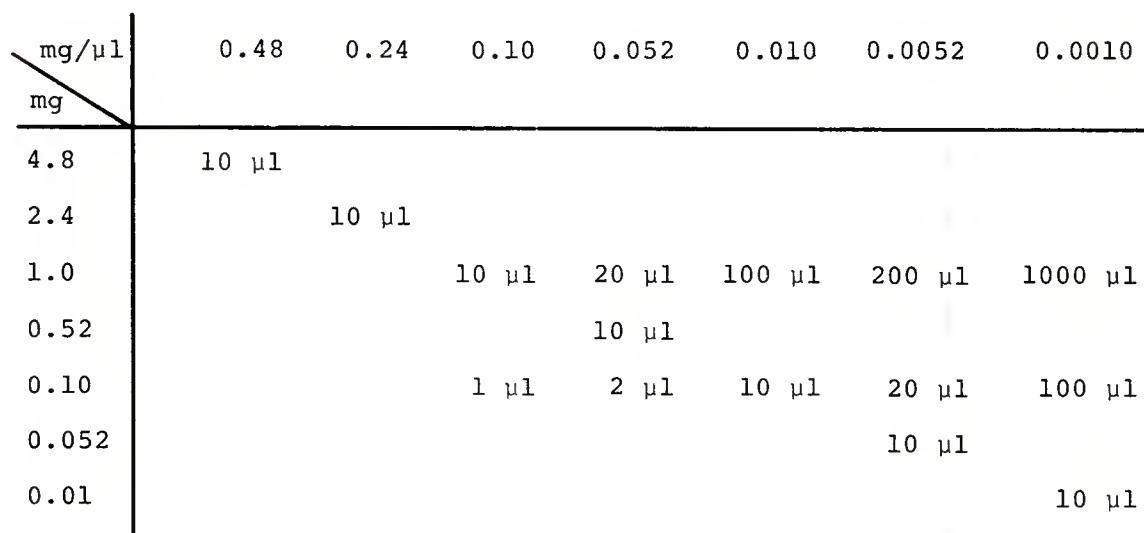


Figure 2. Evaluation of volume and concentration.

Thus, a sample volume of less than $100 \mu l$ and a sample size of between 0.05 and 0.5 mg was selected for all future work.

Calibration

One of the goals of this project was to rapidly determine the molecular weight and molecular weight distribution. Since gel permeation chromatography separates materials by their molecular size, a correlation between the size of a polymer molecule and its molecular weight is necessary. Several methods are available to accomplish this. Some of these include the Q factor method, and the universal calibration method. In the Q factor method, the GPC columns are first calibrated with narrow distribution polymers of known molecular size. Then a well characterized sample of the desired polymer is analyzed and the elution volume of the peak is equal to the number average molecular weight. The ratio of the molecular weight of this polymer to the molecular size of the first is the Q factor. If the second polymer is linear and uniform then by multiplying the molecular size for each of the first polymers by the Q factor, the molecular size calibration is converted to a molecular weight calibration for the desired polymer.

To this end the selected column combination was calibrated to molecular size using narrow distribution polystyrene standards. The elution volumes and molecular sizes are listed in Table 2.

Since no standard is currently available for hydroxy terminated polybutadiene, it was necessary to determine experimentally the number average molecular weight of a sample. To do this, a Mechrolab vapor pressure osmometer, model 301, was used. Unfortunately, the data obtained on this instrument was not reliable and an alternative procedure for calibration was required.

Grubisic, et. al., in "Polymer Letters," Vol. 5, 753 (1967), discussed in detail the "universal calibration" of polymers. This calibration procedure involves calibrating with a polymer of known molecular weight and intrinsic viscosity. A plot of $\log M_w[M]$ versus elution volume should give one straight line for all polymers. Several polystyrene standards have been characterized fully and are described by Cazes and Dobbins in "Polymer Letters," Vol. 8, 785 (1970). The elution volumes were determined for these standards on the column train and the data can be found in

TABLE 2. ELUTION VOLUMES FOR $10^5 + 10^4 + 500 + 100$ \AA^0
COLUMN TRAIN

<u>Material</u>	<u>Size</u> \AA^0	<u>V_i</u> (ml)
Polystyrene	20200	21.58
Polystyrene	9800	22.57
Polystyrene	4160	24.38
Polystyrene	2360	24.92
Polystyrene	1220	25.85
Polystyrene	480	27.67
Polystyrene	117	30.42
C_{34}	43.8	33.0
C_{20}	26.3	35.0
Orthodichlorobenzene	7.7	40.75

Table 3. A plot of the data gives a line with two breaks in the slope. This is not unusual for a combination of columns of different selections.

TABLE 3. POLYSTYRENE STANDARDS

<u>Size (A)</u>	<u>Mw (g/mole)</u>	<u>[η] (dl/g)</u>
244	10,300	0.086
480	19,800	0.142
1220	51,000	0.265
2360	97,200	0.424
4160	160,000	0.627
9800	411,000	1.24

The intrinsic viscosities of several lots of HTPB-R45M from ARCO were determined using a Cannon-Ubbelohde 25 viscometer. Each sample was run at least twice. The water bath temperature was 30.0°C and the solvent was tetrahydrofuran. Results are listed in Table 4.

TABLE 4. INTRINSIC VISCOSITIES

<u>HTPB-R45M Lot Number</u>	<u>[η] (dl/g)</u>
303285	0.20323 ± 0.00128
303305	0.20067 ± 0.00006
402195	0.18925 ± 0.00029
708065	0.20176 ± 0.00004
803105	0.21038 ± 0.00025
803175	0.20843 ± 0.00035
803205	0.22022 ± 0.00022

A Hewlett-Packard lab automation system, model 3353 was obtained to simultaneously gather data from the RI and UV detectors and then analyze the data. A computer program was then prepared to manipulate the raw area versus time data and report the molecular weights of each sample. The intrinsic viscosities of each HTPB-R45M sample were given to the computer and the resulting molecular weights are listed in Table 5. A sample of the computer report is given in Figure 3. It should be noted that the standard deviation of the molecular weights is between 4% and 2%. The limit of reproducibility for gel permeation chromatography is generally considered to be about 5%.

TABLE 5. HTPB-R45M MOLECULAR WEIGHTS

HTPB LOT	303285	303305	402195	708065	803105	803175	803205
VPO	2710	2790	2830				
HPLC	2772 _{1%}	2856 _{4%}	2874 _{2%}	2777 _{1%}	2235 _{2%}	2580 _{2%}	2370 _{2%}

The response of the RI was determined for R45M samples dissolved in THF and in toluene. An accurately determined amount of the polymer was injected at least three times and the total polymer areas were averaged. This was performed on samples ranging from 0.24718 mg to 2.4718 mg. A least squares fit of the data gave a straight line with a coefficient of determination of 0.980 through the origin, and a response factor of 2380 counts/mg.

Evaluation of Derivatives for Functionality Determination

To determine the functionality of a hydroxy terminated polymer, the current process reacts a known amount of the polymer with an isocyanate and then determines the UV absorbance of the aromatic derivative. In transferring this concept to the current project, phenylisocyanate was initially examined. The material reacted slowly and was contaminated with several impurities. Distillation of the isocyanate removed most of the impurities but, on standing in a stoppered flask, the impurities returned. Apparently, this is the result of hydrolysis of the isocyanate. Because of the unstable nature of the phenylisocyanate, another derivatizing agent was considered. In liquid chromatography, it is common practice to derivatize

TIME : 13 : 46 HOURS DATE : 7 JUNE 1979
 SAMPLE : HTPB-R45M LOT 303305 DATA IS FROM RI
 FLOW AVE.B-L SLOPE INTERCEPT STD. DEV.
 2.03252 187701. .429418 187638. 3.60844E-02
 ML/MIN AREA CTS. AREA CTS./SL AREA CTS.
 M W D COMPUTED BETWEEN 11.4 MIN. AND 16.6667 MIN.
 V1 - 23.283 ML. V2 - 27.039 ML.
 EL. TIME EL. VOL. MOL. WT. CORR. AREA CUM. %
 11.3333 23.0352 117238. 1536.94 .324799
 12.3333 25.0678 18280.7 6415.5 11.5326
 13.3333 27.1003 4186.2 12663.1 43.0738
 14.3333 29.1328 2013.99 9044.62 80.1977
 15.3333 31.1653 968.929 2163.19 95.7288
 16.3333 33.1978 466.153 889.75 99.5757
 TOTAL AREA MN MW MO
 473196. 2838.41 8160.05 6109.13
 POLYDISPERSITY SKEWNESS ASSYMMETRY
 2.87486 22.1649 8.63397

Figure 3. Gel permeation chromatographic report.

alcohols with 3,5 dinitrobenzoylchloride. This is a strong UV absorber and very reactive to alcohols at elevated temperatures while only reacting slowly with water at room temperature.

A procedure was developed using 3,5 dinitrobenzoyl-chloride with a pyridine catalyst in toluene at 100°C. Under these conditions the polymer can be partially derivatized in 10 minutes. Continued heating of the sample raised the UV absorbance by 20% after 24 hours. This is approximately equivalent to the amount of internal allylic hydroxyls in the R45M polymers. Further work will be necessary to determine the optimized reaction conditions and minimum reaction time to derivatize quantitatively the internal hydroxyl groups.

CONCLUSION

A procedure for determining the molecular weight distribution has been developed for use on a high performance liquid chromatograph using steric exclusion (GPC) columns. The system has been calibrated using the "universal calibration" procedure and polystyrene standards. The accuracy and precision of this procedure has been determined and is very satisfactory indicating potential use as a standard testing technique for selection of polymer lots.

A sample chromatogram of the derivatized HTPB-R45M including the RI and UV traces is given in Figure 4. The fact that the two normalized traces do not exactly fit each other is strong evidence that derivatization of the hydroxyl groups will eventually lead to a rapid and simple procedure for determination of the functionality distribution of the polymer.

Further work is required to optimize the derivatization procedure using 3,5 dinitrobenzoylchloride. Phenylisocyanate was determined to be an unacceptable derivatizing agent due to its slow reactivity. Another more reactive isocyanate, toluene sulfonylisocyanate, will be evaluated in the next year's effort and the procedure will be expanded to other hydroxy terminated polymers.

RECOMMENDED IMPLEMENTATION

It is recommended that the planned second year's effort be accepted and funded to complete the development of the procedure and evaluate the accuracy of the technique and the extent of its applicability to other polymers than HTPB-R45M.

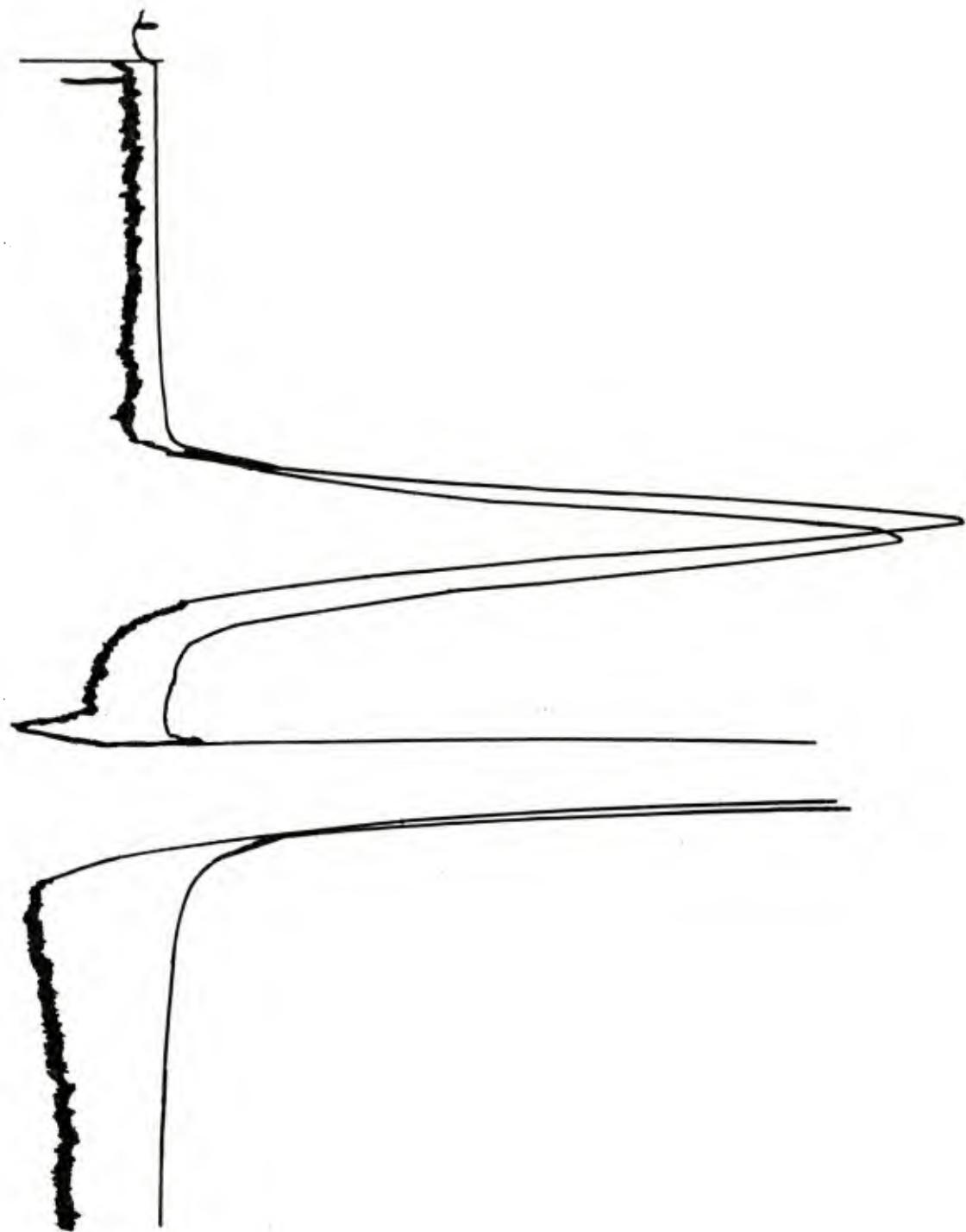


Figure 4. Sample chromatogram of the derivatized HTPB-R45M.

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